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Permittivity of biodiesel-rich blends with fossil diesel fuel: Application to biodiesel content estimation

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HIGHLIGHTS

- Permittivity of biodiesel-rich blends was determined with an uncertainty below 0.3%.
- At each composition, permittivity decreases linearly with temperature.
- At constant temperature, permittivity increases linearly with biodiesel content.
- Biodiesel content is estimated from permittivity and temperature measurements.
- The RMS uncertainty of biodiesel content estimation is below 1.5%.

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ABSTRACT

The relative permittivity of biodiesel-rich blends, from pure biodiesel to 50% blends with diesel fossil fuel, were determined at temperatures between 303.0 K and 343.0 K (controlled within ±0.1 K). Measurements were made on biodiesel from soybean in the frequency range from 20 kHz to 2 MHz; the relative measurement uncertainty was below 0.3%.

At each composition, experimental values fit very satisfactorily to a linear dependence on temperature. Similarly, at constant temperature, permittivity depends linearly on biodiesel content. From these results, a simple model was proposed to estimate the permittivity of the samples as a function of biodiesel content and temperature. The model parameters were determined from a multiple regression analysis. The RMS uncertainty of the fitting was below 0.7%, for blends with biodiesel concentration $\geqslant$50%. The model was inverted to determine the biodiesel content from permittivity and temperature measurements. The parameters of the inverted model were checked by a multiple regression analysis and the RMS uncertainty of the content determination was below 1.5%.

The results presented in this work show that dielectric measurements are a valuable tool for biodiesel content determination in rich-biodiesel blends from vegetable oils with diesel fossil fuel.

1. Introduction

Biodiesel (BD) is an alternative biofuel obtained from the transesterification of a vegetable oil or an animal fat and a short-chain alcohol [1–3]. Its use is steadily increasing in many countries.

BD may be used in blends with diesel fuel (DF) for Diesel engines. Since for fresh products they are completely miscible, the composition of the blends may be chosen according to engine requirements. It must be remarked that in the case of excessive storage times, precipitates might be observed in biodiesel. All the samples studied in this work had storage times shorter than 3 months and were visually checked.

Usually, the composition of a BD/DF blend is indicated as "*Bx*", where "*x*" represents the percentage of BD (V/V). For example, B100 means pure BD and B0 means pure DF.

Due to environmental and supply concerns, regulations in many countries establish that DF must be blended with a certain amount of BD. Moreover, in many developing countries, small-scale biodiesel production from local feedstocks has become an economically viable alternative for the operation of rural machinery, particularly using biodiesel-rich blends with DF. It must be kept in mind that there are several issues that must be addressed to ensure that this

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is an environmentally sustainable and technologically adequate practice. For instance, the impact on water resources [4] and on engine performance and emissions [5] must be taken into account.

Standard methods for *Bx* determination [6] require expensive equipment and trained personnel, and are not often economically viable for small- and medium-scale production. Clearly, alternative methods for *Bx* determination are of technological and environmental interest. At the present state of electronics technology, dielectric measurements are economical, fast and easily adaptable to real-time monitoring systems. They are successfully used for the production [2] and characterization of BD [2,7], and also to characterize feedstocks from different origins [8,9]. They are also useful to detect alcohol in the light phase after the transesterification process [10], during the purification process and in the final product [11,12], for the characterization of FAME [13] and to distinguish between vegetable oil and the biodiesel obtained from it [14]. Several authors have explored the use of dielectric techniques for biofuel and blends characterization for vehicular applications in literature [15–17].

In this work *Bx* is determined by permittivity measurements in the range from 20 kHz to 2 MHz, for samples containing from 50% to 100% (V/V) of BD at temperatures from 303 K to 343 K. This range of compositions and temperatures is particularly important for rural applications in developing countries, in which biodiesel-rich blends (from local feedstocks) with diesel fossil are frequently used.

The results presented in this paper are relevant for the design and implementation of economical, compact and precise composition measurement systems based on dielectric properties.

2. Samples and methods

2.1. Samples

All the samples were prepared with commercial BD provided by a local producer, and met standards EN 14214 and ASTM D6751 [18,19]. Feedstocks utilized for its production were soybean vegetable oil, methanol and sodium hydroxide as a catalyst. Pure DF samples were also provided by a local producer (YPF) and also complied with international standards [20].

2.2. Measurement system

The relative of permittivity of the samples was determined from isothermal dielectric spectra in the frequency range from 20 kHz to 2 MHz. The measurement cell consists of a pair of platinized platinum electrodes. The temperature of the samples was controlled within ±0.1 K with a thermostat (Lauda). Calibration of the system was carried out utilizing cyclohexane as a reference liquid with an uncertainty below 1%. For a more detailed description of the measurement system, see [21]. The measurement uncertainty of real part of permittivity, $\Delta\varepsilon_r'$, was below 0.3% in all cases. All the uncertainties in this work are expressed as one standard deviation.

3. Theory

3.1. Electrical properties

Electrical properties of a substance can be described in the frequency domain by the complex relative permittivity $\varepsilon_r(\omega,T)$ [2,21,22], that is,

$$\varepsilon_r(\omega,T) = \varepsilon_r'(\omega,T) - i\varepsilon_r''(\omega,T) \tag{1}$$

where ε_r is the complex relative permittivity, ε_r' and ε_r'' are respectively the real and imaginary parts of complex relative permittivity, *T* is the absolute sample temperature expressed in *K* and ω is the angular frequency in rads^{-1}.

Given the high purity of the samples studied in this work, no losses were detected in the frequency range studied. Besides, as it was to be expected, no relaxation effects were observed, so ε_r'' was neglected.

In summary, permittivity of liquids studied in this work was modeled by

$$\varepsilon_r(\omega,T) = \varepsilon_r'(T) \tag{2}$$

In the frequency and temperature ranges studied in this work, the permittivity, ε_r', Eq. (2), at each composition Bx_o fits very satisfactorily to a linear dependence with temperature,

$$\varepsilon_r'(Bx_o,T) = \varepsilon_r'(Bx_o,T_o) + \frac{\delta\varepsilon_r'(Bx_o,T_o)}{\delta T}(T - T_o) \tag{3}$$

In Eq. (3), $\varepsilon_r'(Bx_o,T_o)$ is the relative permittivity of a sample with composition Bx_o, at the reference temperature $T_o = 323.0$ K. The temperature coefficient of the relative permittivity, $\delta\varepsilon_r'/\delta T$, at the reference temperature, T_o, is given in K^{-1}; *T* is the absolute temperature of the sample. This dependence had already been reported in pure biodiesel and vegetable oil samples from different feedstocks [2,8,11–14].

4. Results and discussion

4.1. Electrical properties of biodiesel-rich blends with fossil diesel fuel

Fig. 1 shows a three dimensional plot of the real part of permittivity of the samples as a function of temperature and composition. Uncertainty error bands are smaller than the markers.

From Fig. 1 it may be seen that ε_r' values appear to be located on a well-behaved surface (in a mathematical sense). The surface $\varepsilon_r'(Bx,T)$ may be projected onto the (ε_r',T) and (ε_r',Bx) planes, that is, ε_r' can be plotted as a function of *T*, at a fixed biodiesel content Bx_o and as a function of *Bx*, at a fixed temperature T_o.

Fig. 2 shows experimental values of ε_r' as a function of temperature for all the studied samples (projection onto the (ε_r',T) plane). The symbols correspond to the different compositions. The continuous lines represent the fitting of the linear model, Eq. (3), for each sample.

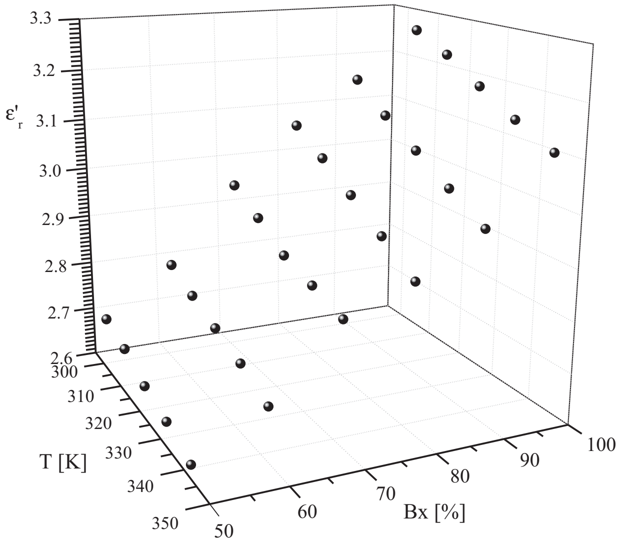

Fig. 1. Experimental values of relative permittivity, ε_r', of BD/DF blends as a function of biodiesel content, *Bx*, and temperature, *T*.

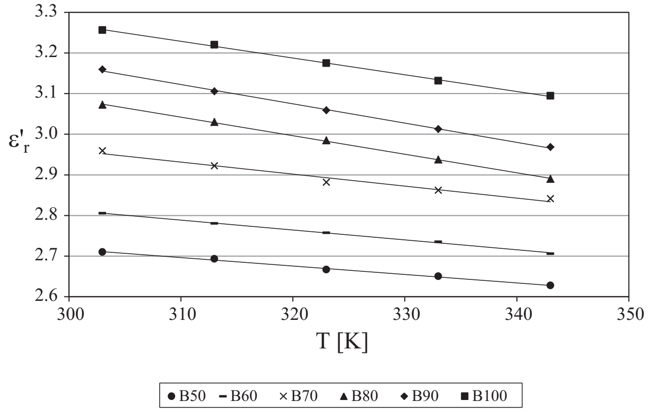

Fig. 2. Projection of the experimental values (symbols) of ε'_r onto the (ε'_r,T) plane. The lines correspond to the fitting of $\varepsilon'_r(Bx,T)$ to Eq. (3) for the studied BD/DF blends.

In Fig. 2 it can be seen that, in all cases, ε'_r decreases linearly with temperature. Moreover, the permittivity of the blends at each temperature increases with *Bx*. Table 1 shows the fitting parameters of Eq. (3) for all the samples studied in this work. The values of $\varepsilon'_r(T_o)$ and $\delta\varepsilon'_r/\delta T$ are given together with their uncertainties, $\Delta\varepsilon'_r(T_o)$ and $\Delta\delta\varepsilon'_r/\delta T$. The RMS uncertainty of the fitting, $\Delta\varepsilon'_r(T)$, and the determination coefficient of the fitting, R^2, are also shown. Reference temperature is T_o = 323.0 K.

From Table 1 it is easy to see that $\varepsilon'_r(T_o)$ increases with *Bx*. Besides, the absolute value of the slope ($\delta\varepsilon'_r/\delta T$) is slightly higher in samples with higher *Bx*. This was to be expected since BD is a more polar substance than DF [8]. The very satisfactory fitting suggests the possibility of estimating the value of *Bx* in BD/DF blends by means of ε'_r measurements.

Fig. 3 shows, in more detail, ε'_r experimental values (symbols) corresponding to a B100 sample. The continuous lines correspond to the linear model (Eq. (3)) and the uncertainty of the fitting is indicated by the dashed lines.

As reported in previous works, a linear dependence with temperature had already been found in pure BD and in vegetable oil samples [2,8,11–13].

Considering the projection onto the (ε'_r,Bx) plane, a first order (linear) model is proposed for $\varepsilon'_r(Bx)$ at each measurement temperature,

$$\varepsilon'_r(Bx, T_o) = \varepsilon'_r(B100, T_o) + \frac{\delta\varepsilon'_r(T_o)}{\delta Bx}[Bx(T_o) - 100] \tag{4}$$

where the fitting parameters are $\varepsilon'_r(B100,T_o)$, the permittivity value of pure BD at temperature T_o, and $\delta\varepsilon'_r(T_o)/\delta Bx$.

Fig. 4 shows experimental values of ε'_r as a function of *Bx* (projection on the (ε'_r,Bx) plane) for all the samples. Symbols indicate experimental data and the continuous lines correspond to the first order model (Eq. (4)).

Table 1
Fitting parameters of Eq. (3): $\varepsilon'_r(T_o)$ and $\delta\varepsilon'_r/\delta T$, their uncertainties $\Delta\varepsilon'_r(T_o)$ and $\Delta\delta\varepsilon'_r/\delta T$, the RMS uncertainty of the fitting, $\Delta\varepsilon'_r(T)$, and the determination coefficient R^2. Reference temperature is T_o = 323.0 K.

Sample	ε'_r (T_o)	$\Delta\varepsilon'_r(T_o)$	$\delta\varepsilon'_r/\delta T \times 10^{-3}$ (K)$^{-1}$	$\Delta\delta\varepsilon'_r/\delta T \times 10^{-5}$ (K)$^{-1}$	$\Delta\varepsilon'_r$	R^2
B50	2.669	0.001	–2.08	8	0.001	0.995
B60	2.757	0.001	–2.44	7	0.001	0.998
B70	2.669	0.001	–2.08	8	0.001	0.995
B80	2.983	0.001	–4.58	6	0.001	0.999
B90	3.061	0.001	–4.75	10	0.001	0.999
B100	3.175	0.001	–4.12	9	0.001	0.998

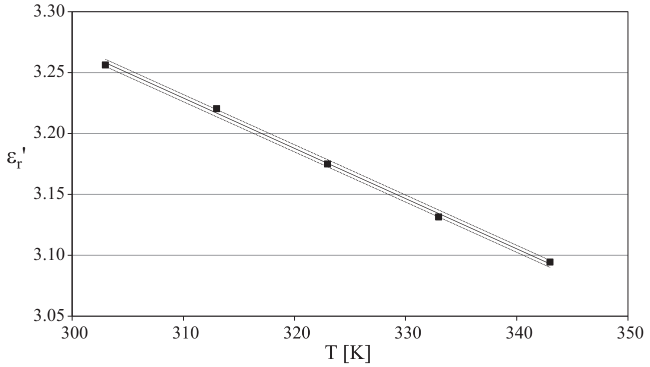

Fig. 3. Relative permittivity, ε'_r, as a function of temperature of a B100 sample.

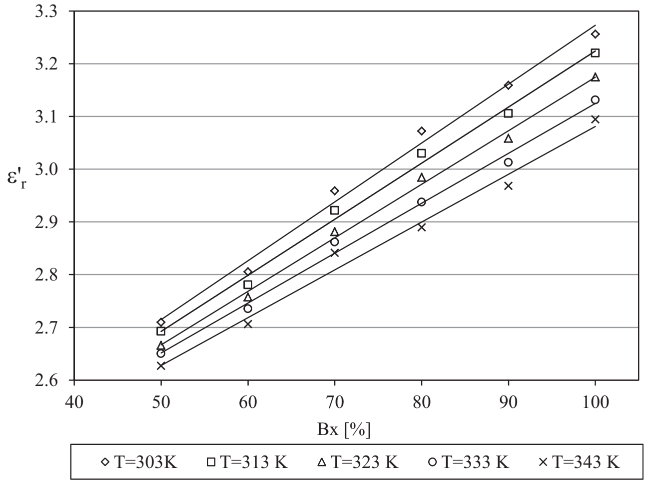

Fig. 4. Relative permittivity, ε'_r, as a function biodiesel content, *Bx*. The lines correspond to the fitting of $\varepsilon'_r(Bx,T)$ to Eq. (4).

Fig. 5 shows in more detail the fitting of experimental values of ε'_r (symbols) to the linear model (Eq. (4)) at the reference temperature, T_o = 323.0 K. The continuous line indicates the fitting and the dashed-lines the limits of the uncertainty band (one standard deviation).

From Fig. 5 it can be seen that the linear model of Eq. (4) fits very satisfactorily to the experimental data.

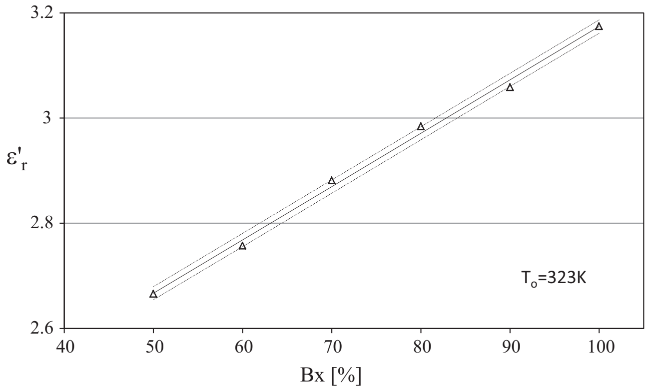

Fig. 5. Relative permittivity, ε'_r, as a function of biodiesel content, *Bx*, at T_o = 323.0 K. The continuous line indicates the fitting and the dashed-lines the uncertainty band (one standard deviation).

Table 2
Fitting parameters of Eq. (4): ε'_r(B100,*T*) and $\delta\varepsilon'_r(T)/\delta Bx$, their uncertainties $\Delta\varepsilon'_r$(B100,*T*) and $\Delta\delta\varepsilon'_r(T)/\delta Bx$, the RMS uncertainty of the fitting, $\Delta\varepsilon'_r$, and the determination coefficient R^2, at each measurement temperature.

Temperature (K)	$\varepsilon'_r(B100)$	$\Delta\varepsilon'_r(B100)$	$\delta\varepsilon'_r/\delta Bx \times 10^{-2}$ (%)$^{-1}$	$\Delta\delta\varepsilon'_r/\delta Bx \times 10^{-4}$ (%)$^{-1}$	$\Delta\varepsilon'_r$	R^2
303	3.27	0.01	1.12	5	0.02	0.992
313	3.22	0.01	1.06	3	0.02	0.994
323	3.17	0.01	1.01	3	0.01	0.997
333	3.12	0.01	9.47	4	0.02	0.994
343	3.08	0.02	9.06	5	0.02	0.987

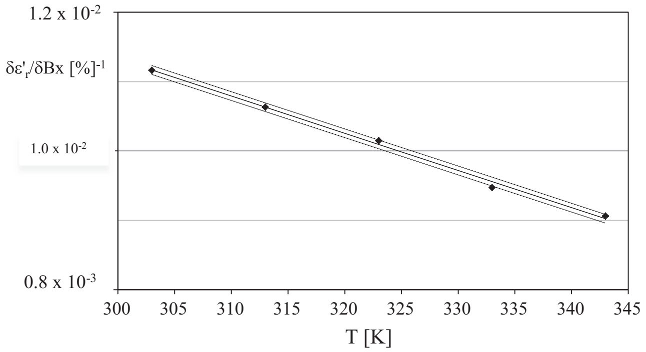

Fig. 6. $\delta\varepsilon'_r(T)/\delta Bx$ as a function of sample temperature, *T*.

Since BD/DF blends below 50% of BD (V/V) are of particular interest for automotive applications, they will be studied in future works.

As it can be seen from previous works, in particular [14], the permittivity values of biodiesel from different feedstocks are very similar to those of biodiesel from soybean. Therefore, the fittings presented in this work for biodiesel from soybean may also be applied, within the stated uncertainties, to biodiesel from other feedstocks. It must be remarked that biodiesel obtained from animal fats was not studied in this work.

It was reported in previous works [2,12] that an adequate treatment, for example filtering followed by drying at 353 K, restores the permittivity of used vegetable oils to values very close to those of the fresh product. In consequence, permittivity values of biodiesel obtained from fresh and treated used vegetable oils will also be very similar. Therefore, the fittings presented in this work may also be applied in this case. Of course, if the treatment of the used oils is deficient, electrical properties of the feedstock may vary in a rather wide range.

Table 2 shows the permittivity of pure BD, $\varepsilon'_r(B100)$, and $\delta\varepsilon'_r(T)/\delta Bx$ together with their uncertainties, $\Delta\varepsilon'_r(B100)$ and $\Delta\delta\varepsilon'_r(T)/\delta Bx$. The RMS uncertainty of the fitting, $\Delta\varepsilon'_r$, and the determination coefficient of the fitting, R^2, are also shown.

As an additional check, it was verified that the permittivity values of pure BD estimated from the linear fitting, $\varepsilon'_r(B100)$, agree very well with previously reported results [2,11–14] from direct measurements of BD permittivity at all the studied temperatures.

From Table 2 it can be noted that, in all cases, the linear model (Eq. (4)) fits the experimental data very well. In fact, the uncertainty of the estimation of ε'_r is below 0.7% in all cases.

Table 4
Fitting parameters of Eq. (8) and RMS uncertainty of the permittivity estimation, $\Delta\varepsilon'_r$.

a	$c \times 10^{-2}$ (%)$^{-1}$	$d \times 10^{-5}$ (%K)$^{-1}$	$\Delta\varepsilon'_r$
2.166	2.52	4.69	0.015

It must be remarked that $\delta\varepsilon'_r(T)/\delta Bx$ depends linearly on temperature. Fig. 6 shows $\delta\varepsilon'_r/\delta Bx$ (symbols) as a function of the measurement temperature. The continuous line corresponds to a linear fitting and the dashed-lines indicate the limits of the uncertainty band (one standard deviation).

Therefore, a first order model is adequate to describe $\delta\varepsilon'_r/\delta Bx$ as a function of temperature, *T*,

$$\frac{\delta\varepsilon'_r(T)}{\delta Bx} = \frac{\delta\varepsilon'_r(T_o)}{\delta Bx} + \frac{\delta^2\varepsilon'_r(T_o)}{\delta Bx\delta T}(T - T_o) \tag{5}$$

where T and T_o the sample and reference temperature respectively, and $\delta\varepsilon'_r(T_o)/\delta Bx$ and $\delta^2\varepsilon'_r(T_o)/\delta Bx\delta T$ are determined by means of a linear regression. Table 3 shows the results of the fitting (Eq. (5)) including the relative uncertainties, the RMS uncertainty of the fitting $\Delta\left(\frac{\delta\varepsilon'_r(T)}{\delta Bx}\right)$ and the determination coefficient, R^2.

The parameters of Eq. (3), $\varepsilon'_r(Bx_o,T_o)$ and $\delta\varepsilon'_r(Bx_o,T_o)/\delta T$, may be now replaced by the first order models given by Eqs. (4) and (5). Therefore, the permittivity of a sample at temperature T and composition Bx ($\geqslant$50%) can now be written as

$$\varepsilon'_r(Bx, T) = \varepsilon'_r(B100, T_o) + \frac{\delta\varepsilon'_r(B100, T_o)}{\delta T}(T - T_o) + \left(\frac{\delta\varepsilon'_r(T_o)}{\delta Bx} + \frac{\delta^2\varepsilon'_r}{\delta Bx\delta T}(T - T_o)\right)[Bx - 100\%] \tag{6}$$

From the above, it is easy to see that, after some algebraic manipulation, Eq. (6) can be expressed as

$$\varepsilon'_r(Bx, T) = a + bT + cBx + dTBx \tag{7}$$

In principle, coefficients *a*, *b*, *c* and *d* could be calculated from Eq. (6) using the values in Tables 1 and 3. As a check, they were also determined by means of a multi-variate regression using all the experimental data and the agreement was very good. Interestingly, results showed that the coefficient *b* is very small, and not statistically significant within the experimental uncertainties, in the range of studied compositions and temperatures. Therefore, for $Bx \geqslant 50\%$, Eq. (7) may be simplified to

$$\varepsilon'_r(Bx, T) = a + cBx + dTBx \tag{8}$$

Table 3
Fitting parameters of Eq. (5): $\delta\varepsilon'_r(T_o)/\delta Bx$ and $\delta^2\varepsilon'_r(T_o)/\ \delta Bx\delta T$, their uncertainties $\Delta\delta\varepsilon'_r(T_o)/\delta Bx$ and $\Delta\delta^2\varepsilon'_r(T_o)/\delta Bx\delta T$, the RMS uncertainty of the fitting, $\Delta\varepsilon'_r$, and the determination coefficient R^2. Reference temperature is T_o = 323.0 K.

$\delta\varepsilon'_r(T_o)/\delta Bx \times 10^{-2}$ (%)$^{-1}$	$\Delta\delta\varepsilon'_r(T_o)/\delta Bx \times 10^{-5}$ (%)$^{-1}$	$\delta^2\varepsilon'_r/\delta Bx\delta T \times 10^{-5}$ (%K)$^{-1}$	$\Delta\delta^2\varepsilon'_r/\delta Bx\delta T \times 10^{-6}$ (%K)$^{-1}$	$\Delta\delta\varepsilon'_r/\delta Bx \times 10^{-5}$ (%)$^{-1}$	R^2
1.01	3	−5.40	2	6	0.996

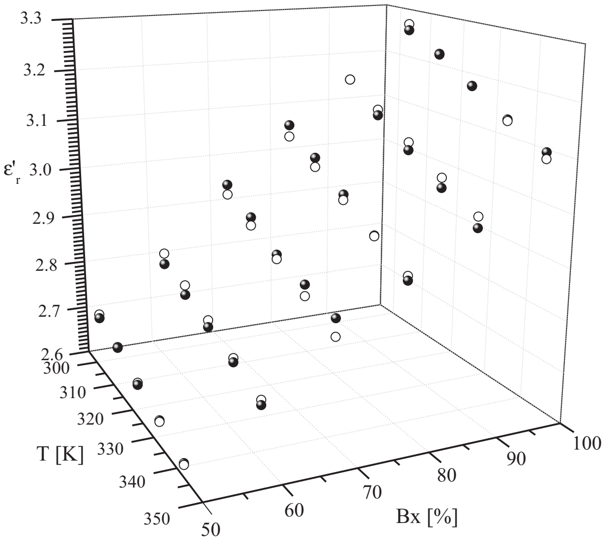

Fig. 7. Permittivity experimental data (black symbols) and estimations from Eq. (8) (empty symbols).

Table 5
Fitting parameters of Eq. (9) and RMS uncertainty of the composition estimation, ΔBx.

a	$c \times 10^{-2}$ $(\%)^{-1}$	$d \times 10^{-5}$ $(\%\mathrm{K})^{-1}$	ΔBx (%)
2.162	2.55	−4.74	1.5

It must be stressed that Eq. (8) may not be applied for blends of lower *Bx*, since it does not reflect the well-known dependence of DF permittivity on temperature [23]. However, for the biodiesel-rich blends studied in this work, this simplification does not introduce statistically significant errors.

Table 4 shows the fitting values of the coefficients in Eq. (8), together with the RMS fitting uncertainty of the permittivity, $\Delta\varepsilon'_r$.

It may be seen that the RMS uncertainty of the permittivity estimation, $\Delta\varepsilon'_r$, is close to the uncertainty of permittivity measurements (≅0.01). Therefore, Eq. (8) is a good approximation to $\varepsilon'_r(Bx,T)$ within the range of measured composition and temperatures. Fig. 7 shows a plot of permittivity experimental values (black symbols) and permittivity estimation from Eq. (8) (empty symbols) as a function of temperature and composition.

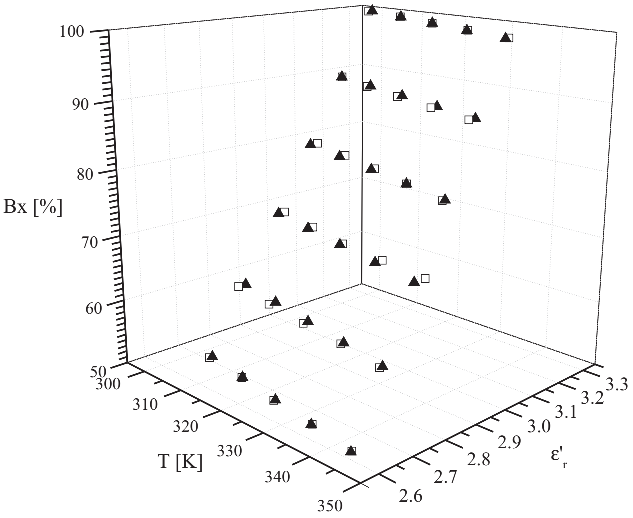

Fig. 8. Experimental values of composition (black triangles) and estimations from Eq. (9) (empty triangles) as a function of temperature and permittivity.

4.2. Estimation of biodiesel content in biodiesel-rich blends

Since the model proposed in Section 4.1 fits experimental data very satisfactorily, the simplified model of Eq. (8) was inverted to determine the biodiesel content of biodiesel-rich blends ($Bx \geqslant 50\%$) from permittivity and temperature measurements,

$$B_x(\varepsilon'_r, T) = \frac{\varepsilon'_r - a}{c + dT} \qquad (9)$$

Clearly, the parameters of Eq. (9) could be obtained from Table 4. As a check, they were also determined by means of a multiple non-linear regression using all the experimental data; the agreement was very satisfactory. Table 5 shows the fitting values of the coefficients and the RMS uncertainty of the fitting of the composition estimation, ΔBx.

As expected, coefficients in Tables 4 and 5 agree very well, and Eq. (9) estimates *Bx* very satisfactorily as a function of ε'_r and *T*, with an RMS uncertainty below 1.5%. Fig. 8 shows the composition of the samples (black triangles) and the estimation from Eq. (8) (empty squares) as a function of temperature and permittivity.

5. Conclusions

The relative permittivity of biodiesel-rich blends with diesel fossil (⩾50% (V/V)) was determined on biodiesel from soybean, in the frequency range from 20 kHz to 2 MHz and at temperatures between 303.0 K and 343.0 K. The measurement uncertainty was below 0.3%.

As it was to be expected, for all the samples studied, the results were practically independent of frequency.

At each composition, the relative permittivity decreases linearly with temperature.

Similarly, at constant temperature, permittivity increases linearly with biodiesel content.

From these results, a simple model was proposed to estimate the permittivity of the samples as a function of biodiesel content and temperature, valid for biodiesel-rich blends (⩾50% (V/V)).

Since the comparison with the experimental data was very satisfactory (the RMS uncertainty of the estimation was below 0.7%), the model was inverted to estimate biodiesel content from permittivity and temperature measurements. As a check, the parameters were independently determined by a multiple non-linear regression analysis using all the experimental data. The RMS uncertainty of the composition estimation was below 1.5%.

The results presented in this work show that dielectric measurements are a valuable tool for biodiesel content estimation in biodiesel-rich blends with diesel fossil fuel.

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